ATTACHMENT A

Protocol for Nitrogen Sample Collection and Analysis for Waste Water Effluent

Approved Analytical Methods (from Table 1 B of Part 136 per the 2012 Method Update Rule): (laboratory must be certified for any method performed)

Total Kjeldahl Nitrogen (TKN):

Manual digestion and distillation or gas diffusion followed by any of the following	SM4500-Norg B-97 or C-97 and SM4500-NH3 B-97.		ASTM D3590-02 (06) (A)	I-4515-9145
Titration	SM4500-NH3 C-97		ASTM D3590-89, 02 (A)	973.48.3
Nesslerization			ASTM D1426-08 (A)	
Electrode	SM4500-NH3 D-97 or E-97		ASTM D1426-08 (B)	
Semi-automated phenate	EPA 350.1 Rev. 2.0 (1993)		SM4500-NH3 G-97 or H-97	
Manual phenate, salicylate, or other substituted phenols in Berthelot reaction based methods	SM4500-NH3 F-1997			
Automated methods for TKN that do not require manual digestion				
Automated phenate, salicylate, or other substituted phenols in Berthelot reaction based methods colorimetric (auto digestion and distillation)	EPA 351.1 (1978)			I-4551-788
Semi-automated block digestor colorimetric (distillation not required)	EPA 351.2, Rev. 2.0 (1993)	SM4500-Norg D-97	ASTM D3590-02 (06) (B)	I-4515-9145

Nitrate + Nitrite (NO3 + NO2):

Cadmium reduction, Manual		SM4500-NO3 E-00	ASTM D3867-04 (B)	
Cadmium reduction, Automated, or	EPA 353.2, Rev. 2.0 (1993)	SM4500-NO3 F-00	ASTM D3867-04(A)	I-4545-852
Automated hydrazine		SM4500-NO3 H-00		
Ion chromatography	EPA 300.0, Rev. 2.1 (1993) and EPA 300.1, rev. 1.0 (1997)	SM4110 B-00 or C-00	ASTM D4327-03	993.303
CIE/UV		SM4140 B-97	ASTM D6508-00 (05)	ASTM D6508, Rev. 2

Sample Collection: The Maine DEP is requesting that nitrogen analysis be conducted on composite effluent samples, unless a facility's Permit specifically designates grab sampling for this parameter. Facilities can use individual collection bottles or a single jug made out of glass or polyethylene. Bottles and/or jugs should be cleaned prior to each use with dilute H_2SO_4. This cleaning should be followed by several rinses with distilled water. Commercially purchased, pre-cleaned sample containers are an acceptable alternative. The sampler hoses should be cleaned, as needed.

Sample Preservation: During compositing the sample must be at 0-6 degrees C (without freezing). If the sample is being sent to a commercial laboratory or analysis cannot be performed the day of collection then the sample must be preserved using H_2SO_4 to obtain a sample pH of <2 su and refrigerated at 0-6 degrees C (without freezing). The holding time for a preserved sample is 28 days.

Laboratory QA/QC: Laboratories must follow the appropriate QA/QC procedures that are described in each of the approved methods.

Sampling QA/QC: If a composite sample is being collected using an automated sampler, then once per month run a blank on the composite sampler. Automatically, draw distilled water into the sample jug using the sample collection line. Let this water set in the jug for 24 hours and then analyze for total nitrogen. Preserve this sample as described above.

ATTACHMENT B

Protocol for Total Phosphorus Sample Collection and Analysis for Waste Water and Receiving Water Monitoring Required by Permits

Approved Analytical Methods: EPA 200.7 (Rev. 44), 365.1 (Rev. 2.0), (Lachat), 365.3, 365.4; SM 3120 B, 4500-P B.5, 4500-P E, 4500-P F, 4500-P G, 4500-P H; ASTM D515-88(A), D515-88(B); USGS I-4471-97, I-4600-85, I-4610-91; OMAAOAC 973.55, 973.56

Sample Collection: The Maine DEP is requesting that total phosphorus analysis be conducted on composite effluent samples, unless a facility's Permit specifically designates grab sampling for this parameter. Facilities can use individual collection bottles or a single jug made out of glass or polyethylene. Bottles and/or jugs should be cleaned prior to each use with dilute HCL. This cleaning should be followed by several rinses with distilled water. Commercially purchased, pre-cleaned sample containers are an acceptable alternative. The sampler hoses should be cleaned, as needed.

Sample Preservation: During compositing the sample must be at 0-6 degrees C (without freezing). If the sample is being sent to a commercial laboratory or analysis cannot be performed the day of collection then the sample must be preserved using H_2SO_4 to obtain a sample pH of <2 su and refrigerated at 0-6 degrees C (without freezing). The holding time for a preserved sample is 28 days.

Note: Ideally, Total P samples are preserved as described above. However, if a facility is using a commercial laboratory then that laboratory may choose to add acid to the sample once it arrives at the laboratory. The Maine DEP will accept results that use either of these preservation methods.

Laboratory QA/QC: Laboratories must follow the appropriate QA/QC procedures that are described in each of the approved methods.

Sampling QA/QC: If a composite sample is being collected using an automated sampler, then once per month run a blank on the composite sampler. Automatically, draw distilled water into the sample jug using the sample collection line. Let this water set in the jug for 24 hours and then analyze for total phosphorus. Preserve this sample as described above.

ATTACHMENT C

Whole Oceans Estimated Chemical Annual Usage (Discharged to Outfall 003)

Fungicides, Topical Bactericides, Parasiticides:

- Povidone iodine (iodophor): Active ingredient 10% polyvinylpyrrolidinone. Typical dose range for egg disinfection 50-100 ppm.
- Hydrogen peroxide (35% Perox-Aid): Active ingredient 35% hydrogen peroxide. Used for control of fungus on eggs/fish and potentially for bacterial gill disease on fish. Typical dose range between 100-1000 ppm depending on use.
- Chloramine-T (Halamid): Active ingredients N-chloro, p-toluenesulfonamide and sodium salt trihydrate. Used for control of bacterial gill disease. Typical dose range 12-20 ppm.
- Potassium permanganate: Considered as 97% active. Used for control of certain parasites and fungal infections in younger fish life-stages. Typical dose range 1.5-2.5 ppm.

Antibiotics:

- Terramycin® 200 (oxytetracycline dehydrate, 44% active): Used in accordance with label for a maximum of 3.75 g active oxytetracycline/100 lb fish/day as an in-feed treatment for susceptible bacterial infections.
- Aquaflor® (florfenicol; 50% active): Used in accordance with label with maximum of 15 mg/kg fish/day as an in-feed treatment for susceptible bacterial infections.
- Romet® 30/Romet® TC (sulfadimethoxine/ormetoprim, 30% active or 20% active, respectively): In accordance with label, 50 mg/kg fish as an in-feed treatment for susceptible bacterial infections.

Disinfectants:

- Sodium hypochlorite (bleach): Active ingredient: 8% sodium hypochlorite in concentrated form. Typically used at 100-1000 ppm for general cleaning/disinfection. Approximate annual use: 250 gallons of 1:100 diluted form.

Other Therapeutants:

- Sodium chloride: Discharge of up to 35 kg NaCl/day for periodic treatment of fish in nursery units, and discharge of up to 42,350 kg/day for maintaining salinity in growout systems.
- Calcium chloride [amount TBD]